DESIGN OF CENTRIFUGAL CASTING EXPERIMENTAL SETUP FOR FABRICATION OF ALUMINA TUBE

A THESIS IN THE PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF BACHELOR OF TECHNOLOGY

SUBMITTED BY:-
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CERTIFICATE

This is to certify that the project report entitled "**Design of Centrifugal Casting Experimental Setup for Fabrication of Alumina Tube**", being submitted by Mr. Abhay Kumar, Department of Ceramic Engineering, National Institute of Technology Rourkela, as partial fulfilment of the requirements for the Degree of **Bachelor of Technology in Ceramic Engineering** is a record of bona fide work carried out by him, under my supervision. The results of investigations enclosed in this report have been verified and found to be more than satisfactory.

The results embodied in this Project Report have not been submitted to any other University or Institute for the Award of any other Degree or Certificate.

Date- 13-05-2013

Skpratihar 13/5/13

Dr. S.K. Pratihar

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ABSTRACT

Centrifugal casting is one of the advanced casting techniques widely used in metallurgical industries. It has been reported that centrifugal technique is very useful for production of functionally graded porous membranes for gas permeable applications. Studies on different centrifugal casting machine design suggest that the centrifugal casting machine consists of a high speed motor (capable of rotating at high speed greater than 12000 rpm) equipped with a suitable sample holder capable of rotation around the same axis of the motor. Laboratory centrifuge motor has speed in the same range as that required for a centrifugal casting machine. Thus has a potential to be used in design of centrifugal casting experimental set up.

In the present work, an attempt has been made to design an experimental set up for centrifugal casting of ceramic slurry using the motor of a laboratory centrifuge. However it make it feasible one need to design and fabricate a suitable rotor head capable to hold tightly the casting mould while rotating in the same axis of the motor. Thus, the present study aims to design and fabricate a suitable rotor head in order to make the centrifugal casting technique viable. The rotor head has been designed and fabricated in such ways that it can tightly fitted with motor shaft and hold the mould during rotation without any appreciable vibration. An attempt has also been made to cast alumina tube using the experimental set up thus designed. In order to fabricate the alumina tube, aqueous alumina slurry has been prepared with the addition of suitable electrolyte and binder. Microstructure of sintered body has also been studied.

CHAPTER-1

INTRUDUCTION

Slip casting is a traditional process for making ceramic body. This process can be used for forming a complex shape body. The common casting methods are solid casting, drain casting, battery casing to name a few. With the help of solid casting one can make a solid body, whereas using drain casting method one can make hollow body such as crucible, tube etc. casing process involves preparation of a stable ceramic slurry and consolidation of the slurry by dewatering through a porous mould. The dewatering process takes the advantages of capillary forces of the porous mould. Centrifugal Casting is a one type drain casting method in which one uses the advantage centrifugal forces for dewatering the slurry.

Centrifugal casting is one of the advanced casting techniques widely used in metallurgical industries. However, it is rarely used in ceramic. Few literatures are available on fabrication of ceramic body using centrifugal casting technique. It has been reported that centrifugal technique is very useful for production of functionally graded porous membranes for gas permeable applications. A detailed study of the principle and operations of centrifugal casting machines available commercially suggests that there exist two types of centrifugal casting machine designs. These designs are based mainly on the operating of the rotating axis. They are i) horizontal centrifugal casting and ii) vertical casting machines. Study of different design suggest that for centrifugal casting machines are consists of a high speed motor equipped with a suitable sample holder capable of rotation around the same axis of the motor.

The present study aims to develop a vertical centrifugal casing set up for fabrication of ceramic body. In this purpose attempt has been made to use the motor a laboratory centrifuge as the rotor. A suitable sample holder or rotor head has been design. This sample holder is capable to hold the impervious mould tightly during rotation. Attempt has also made to prepare a stable aqueous alumina slurry and fabrication of alumina tube using the in house made centrifugal setup.

Following are the brief introduction to different chapters which have been written in the thesis. Chapter 2 gives a brief survey of the literature, the methods followed, the mechanism and the advantages of centrifugal casting. Chapter 3 gives us an insight into the objective behind designing of a setup for centrifugal casting. Chapter 4 details out the Experimental procedures followed during the design of the sample holder, the set-up for the centrifugal casting, the preparation and characterization of the stable alumina slurry. Chapter 5 discusses at length the results obtained in the characterization of the stable alumina slurry.

CHAPTER-2

2.1 CENTRIFUGAL CASTING

Centrifugal casting is an advanced slip casting technique; this method can be used for casting a body which has axial symmetry. This method is commonly used in casting of molten metal, but it can be used in casting of slurry which contains ceramic powder. The method involves pouring of molten metal into a cylindrical mould rotating about its axis of symmetry. The mould is kept rotating till the metal has solidified. For molten metal case mould material is stainless steel, cast iron and graphite. But in the case of ceramics, mould material may be stainless steel, Teflon and nylon.

2.1.2 Principle

When a body moves on a circular path as shown in Fig.2.1, two type of force acting on it namely Centripetal Force - this force acted along the radius towards the centre and Centrifugal Force - this force is acts outward force that draws a rotating body away from the centre of rotation. Fig. 2.1. Shows the different forcess acting on a rotating body in a circular path.

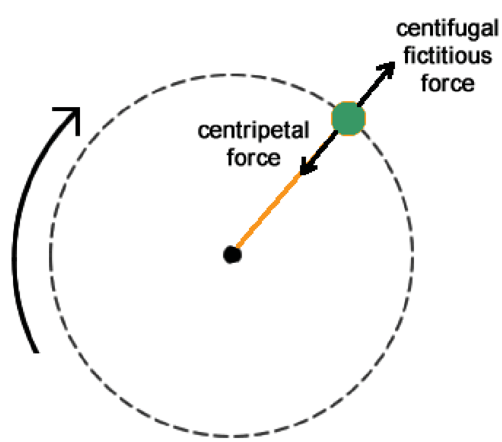


Fig.2.1 Schematic of different forces acting on a rotating body.

This concept is used in centrifugal operation. When a mould filled with slurry is rotate at high speed then there are two forces acting on the particle if the centrifugal force is greater than the centripetal force the particle stick on the wall of the mould surface. These forces are velocity and mass dependent. For high mass forces is high.

2.1.3 Slip casting versus Centrifugal casting

Ceramics fabricated by casting process involves preparation of stable slurry with addition of different additives and ceramic powder in a liquid medium. Removal of liquid from the slurry, to form a powder compact which is subsequently dried and sintered at high temperature in order to get a ceramic body. The most commonly used liquid for the preparation of the slurry is water. Hence the slurry is called aqueous slurry and the removal of liquid from the slurry is commonly known as dewatering. Dewatering in conventional slip casting technique takes the advantage of the capillary force arising from the presence of the porosity in the porous mould. Aqueous slurry in contact with the surface of the porous mould experiences the capillary force there by water from the slurry starts penetrating in the porous mould, leaving the suspended ceramic particle on the surface of the mould. As a result a cast layer is formed at the mould-slurry interface. With the advance of time thickness of the cast layer is gradually increases. When required thickness of the cast layer is achieved the excess slurry is drained out from the mould, which leads to fabrication of hollow ware. This technique is known as drain casting technique.

In case of solid casting, the slurry is allowed for dewatering until the cast layer thickness achieves the shape of the body to be cast.

Centrifugal casting takes the advantages of centrifugal forces for the consolidation of particle present in the slurry. Hence the solid-liquid separation in centrifugal casting occurs by centrifugal force as compare to the capillary force in case of slip casting. The magnitude of the centrifugal force can be tailored by changing the rotational speed of the centrifugation. The magnitude of the centrifugal force is an order of magnitude higher than the capillary force. This drastically reduces the casting time. However, only symmetrical hollow ware can be fabricated by this type of technique.

2.1.4 Advantage of centrifugal casting

- Axial symmetrical body can be fabricated easily.
- Inner part of the body has highly dense
- Body has non-uniform microstructure and a pore gradient through out of the body so it can be used in membrane application

2.1.5 Different types of centrifugal casting methods

I. Horizontal centrifugal casting: -

The method casting involves rotating the mould in a horizontal axis. A typical horizontal centrifugal casting machine is shown in Fig.2.2. Horizontal centrifugal casting is preferred for the tube geometry as the diameter for such geometry is less than their length.

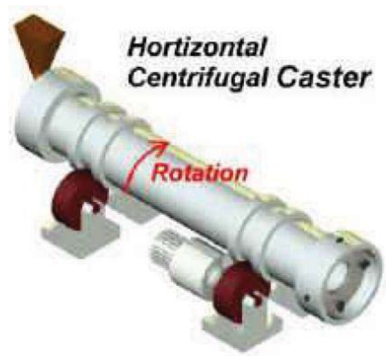


Fig.2.2 Horizontal centrifugal casting machine [1].

II. Vertical centrifugal casting: -

This type of casting is followed for cylinder type bodies having ring geometry. In this method the mould is rotated in a vertically axis. A typical vertical centrifugal casting machine is shown in Fig. 2.3. This method is more suitable for these bodies when the diameter is greater than their length.

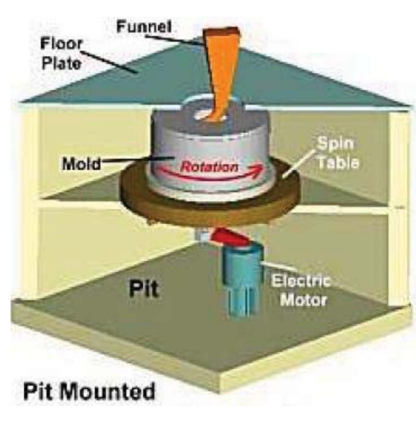


Fig. 2.3 Vertical centrifugal casting machine [2].

2.2 LITERATURE SURVEY

Alumina tubes of different diameters have been fabricated by centrifugal casting from weakly coagulated slurry [3]. It has been reported that the microstructure of the tube was non-uniform and there exists a porosity gradient along the thickness of the sample. It has been correlated with the different settling during casting. It has also been reported that the porosity, pore size distribution and the permeability of the tube could be controlled through the variation of stating alumina powder and sintering schedule. The study suggests that centrifugal casting as promising technique for alumina membrane fabrication.

Porous alumina tube for gas permeation has been fabricated by centrifugal casting technique using different alumina powder with narrow powder size distribution [4]. It has been reported that the pore gradient observed across the wall thickness of the cast body depend on slurry character. Polymethylmethacrylate (PMMA) has been used as pore former in the study in order to develop microspores in the cast body. Microstructural study revealed that it consists of bimodal pore size distribution. It has been attributed that the larger pores in the mirometric range are formed due to the bun out of the pore former and the sub micrometric pores are formed either due to the low packing density of the centrifugal casing process or low sintering temperature of the tube. Bimodal pore has been reported to be very useful to control the air permeability. The study reports that pore graded tube showed higher strength compare to homogenous tube with same inner wall porosity.

Effect of particle size distribution on the green density of slip cast alumina body has been studied by Tari et al. [5]. It has been reported that a bimodal distributed alumina powder could result in a very good green density of the cast body when the end components of the bimodal distributed powder are mixed in a specific size and volume ratio. The increase in green density of the cast body has been correlated with the lowering of the viscosity of the slurry. It has also been reported that the green density of the cast body increases with the increase in particle loading of the slurry.

Alumina body has been fabricated using centrifugal casting technique. The effect of particle size and impurity level of alumina powder on the properties of cast bodies has been studied [6]. It has been reported that the powder with smaller particle size and lower impurity could be dispersed to low solid loading with small amount of addition of dispersants compared to powder with large particle size and high impurity level because smaller particle produces higher inter particle attraction forces which lead to agglomeration. Studies have suggested that with increase in solid loading the packing density of the body increases. It has been observed that mass segregation in the compact is negligible when solid loading is high in the slip. It has been concluded that green body has a packing density gradient and density decreases linearly from bottom to top of the body. The gradient observed is smaller in the case of the fine powder because the finer particle can be packed into voids. Body prepared with fine powder have shown lower sintering temperature due to their high surface area and good mechanical properties compared to coarse powder.

Alumina-zirconia composite body has been fabricated using centrifugal casting technique with different solid loading of alumina, by adding 15 wt% (ZrO_2, 3 mol% Y_2O_3) and small amount of poly-carboxylic ammonium as dispersants [7]. It has been observed that the dispersing agent greatly influences viscosity of the slurry. The report suggested that the green and sintered density of the composite increases with the increase in the solid content. The study concludes by suggesting that phase segregation could be eliminated when solid content is 78 wt.% in the slurry.

α-Alumina ceramic components showed high reliability at high bend strength levels fabricated via the centrifugal casting technique [8]. It has been observed that the wet processing of ceramic powders and subsequent centrifugal casting of high-solid-content suspensions provided a route to produce ceramic components that showed better particle packing and a smaller number and size of flaws than dry processed components. The reported values of green and sintered densities were high. Microstructural analysis revealed that the microstructures were homogeneous. Values observed for the four-point-bend strength were found to be 540 MPA and a reliability characterized by Weibull modulus was found to be m = 23.9. the observed results has been correlated with the speciality of fabrication technique adopted in this study.

Massive and near-net-shape TZP ceramic parts with better particle packing than isostatically pressed TZP has been fabricated using centrifugal casting technique [9]. Casting slip used in this study contains very high particle loading. It has been observed that the number of flaws, their sizes and pores were much smaller in the green body. This in turn leads to a high green density of the body. As a result high sintered densities have been achieved. Centrifugally cast parts with theoretical densities (99-8% TD) have been achieved at temperatures as low as 1300°C. Centrifugally cast sintered compacts showed a finer and more homogeneous microstructure than isostatically pressed TZP. The reported values for fracture toughness, after post-sintering annealing were as high as ~13MPa/m. It has also been reported that the centrifugally cast materials exhibited a narrow grain size distributions than the dry isostatically pressed materials.

Tubular ceramic membrane has also been fabricated and studied by Bissett et.al. [10]. It has been demonstrated that membrane support with graded porosity could be fabricated by the centrifugal casting technique. Low temperature sintered supports showed high porosity, high water permeability and low tensile strength. This has been attributed with the porosity of the samples. Effect of starting powder particle size has been reported in this investigation. It has been reported that the pore size and pore size distribution of the sintered body significantly influenced by the starting powder particle size and sintering temperature.

CHAPTER-3

OBJECTIVES

Centrifugal casting is an advanced technique of casting, which is widely used in metallurgical industry. However the technique is rarely used in the fabrication of a ceramic body. A few literatures are available for the fabrication of ceramic bodies by the centrifugal casting technique. Study of the different designs revealed that centrifugal casting machine consists of two parts; a high speed motor capable of roaring at a speed up to 12000 rpm and a rotor head capable of holding casting mould tightly during rotation without any appreciable vibration. Thus the present study aims to achieve the following:

i) Modification of laboratory centrifuge (which is equipped with a high speed motor capable of rotating at a speed more than 12000 rpm) with a suitable rotor head.

ii) Design of a vertical rotor head of suitable geometry which is capable of hold the casting mould tightly during centrifugal casting.

iii) Preparation of a casting slip with suitable additives and fabrication of ceramic body using the centrifugal casting setup and drain casting.

CHAPTER-4

EXPERIMENTAL

4.1 Sample holder design

Centrifuge tube has been used as a mould in the present study. The 3D isometric view of the centrifuge tube is shown in Fig.4.1. The dimensions of the centrifuge tube have been taken account in the design of rotor head.

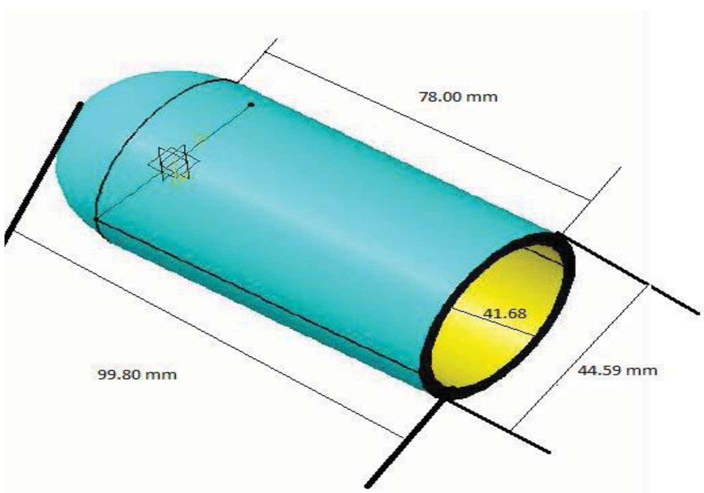
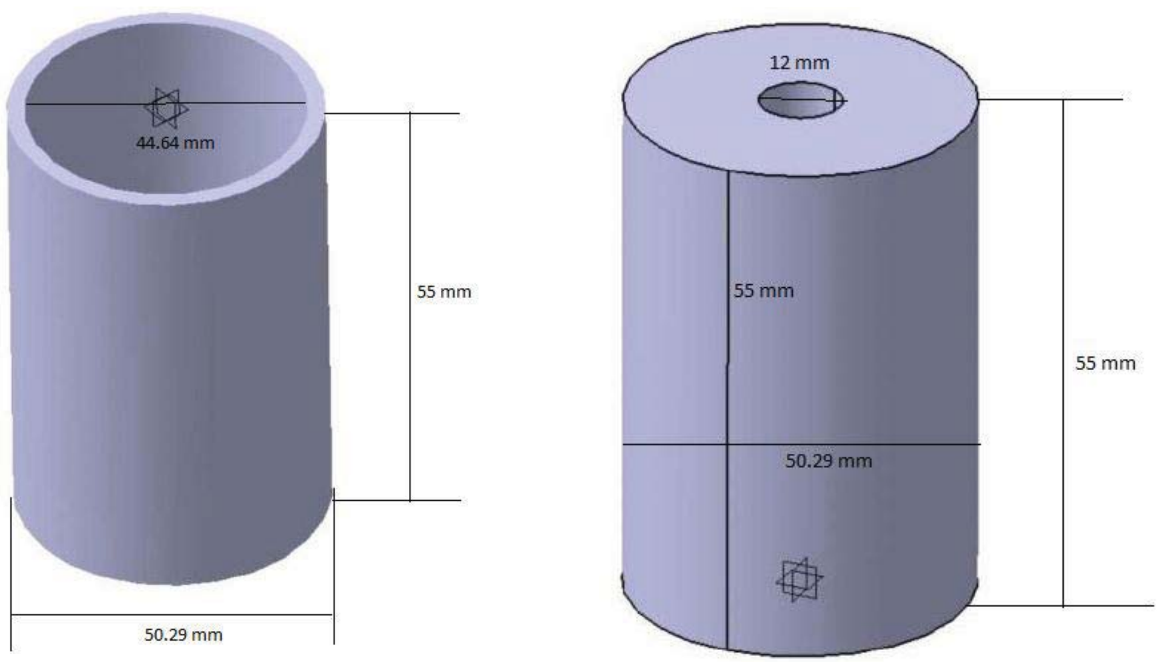


Fig. 4.1 Isometric view of the mould to be used in centrifugal casting technique.

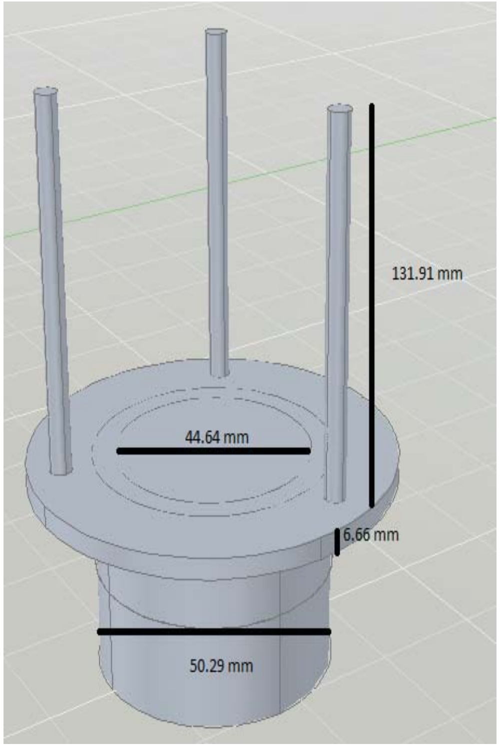
Rotor head has to be fitted tightly with the shaft of the centrifuge motor. It will be capable of holding the mould during the centrifugal casting operation without any significant vibration. Brass is used to fabricate the rotor head due its easy machinability. of the For the design of a mould holder we take a brass rod of diameter 50 mm and length 100 mm. Brass has good mechanical strength and cutting with the lathe machine is easy. Accordingly cuts the brass by the lathe machine in correspondence to the size of mould and size of motor shaft.

Initial design of rotor head has been shown in Fig.4.2, wherein (a) showed the hole for mould fitting and (b) showed the hole to be fitted with the motor shaft. The threads in shaft side are made anticlock wise, because motor rotates clock wise. These types of arrangement will tight the rotor head during centrifugal operation. Care has not be taken for the vertical movement of slurry during centrifugal operation in this design. Hence attempt to cast body with this rotor head lead to spillage of slurry due to vertical movement. In improved design a ring is fitted in the previous mould which will stop the vertical movement of the slurry during centrifugal operation. The ring contains three threaded column as shown in Fig.4.3(a). With the help of this columns and a metal ring as shown in Fig.4.3(b) the mould can be tight fitted so that slurry cannot spill out during rotation as shown in Fig.4.3(c).

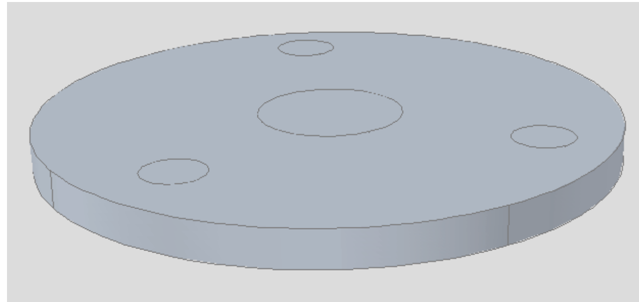


(a) **(b)**

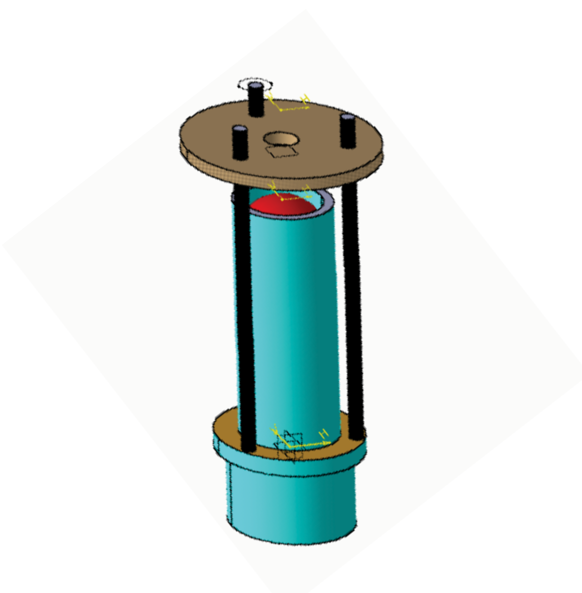
Fig.4.2 Initial rotor head design (a) showed the hole for mould fitting and (b) showed the hole to be fitted with the motor shaft.



(a)



(b)



(c)

(d)

Fig. 4.3 Improved rotor head design (a), metallic ring to hold the mould tightly with the rotor head (b), complete rotor head with mould fitted in it (c) and actual rotor head (d).

4.2 Experimental setup

Schematic centrifugal casting experimental setup has been shown in Fig.4.4. This set up is consists of the following parts: Mould, rotor head capable of holding the mould rigidly during casting and a high speed motor (capable of rotating at a speed above 12000 rpm).

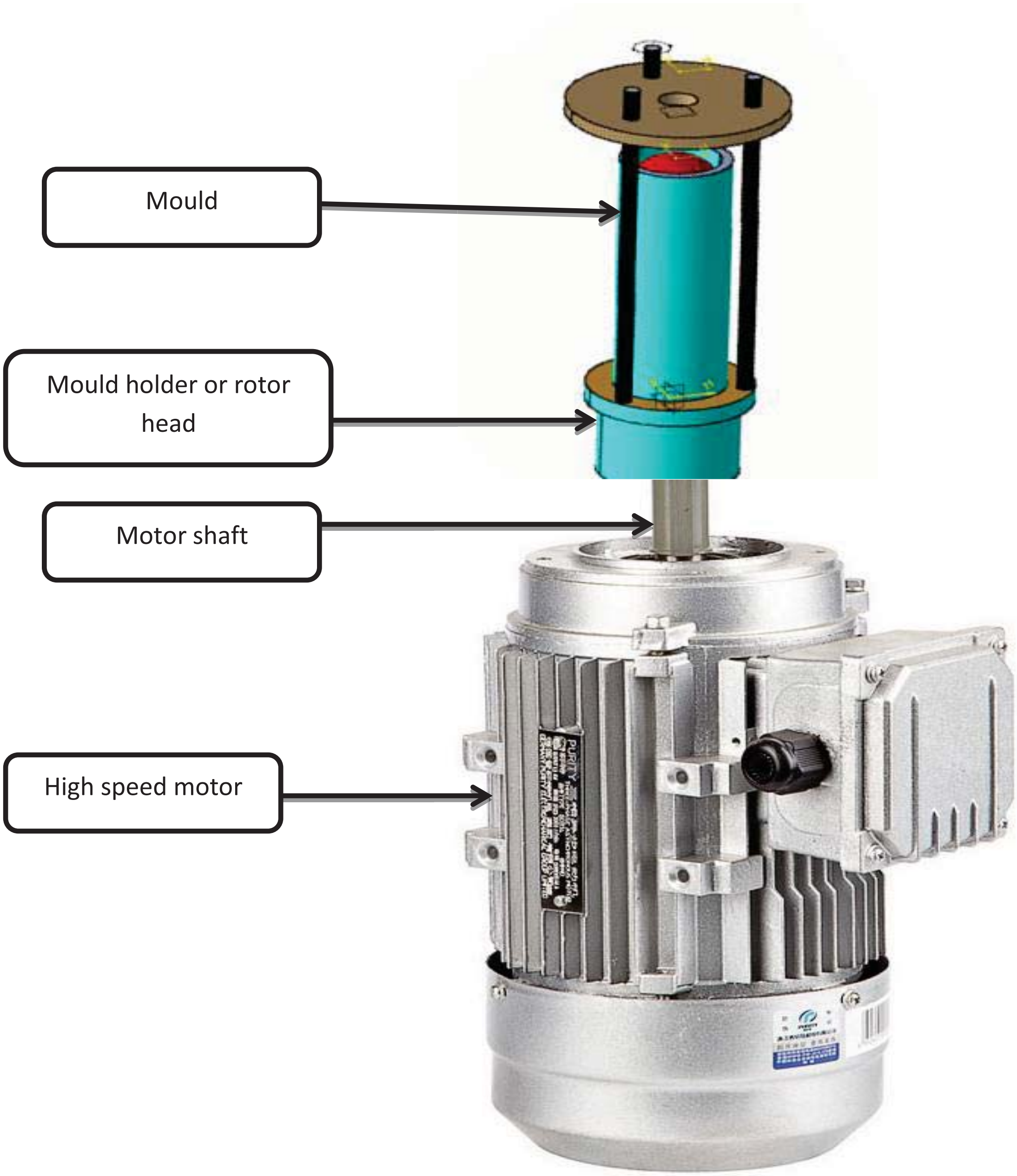


Fig. 4.4 Experimental set up for centrifugal casting of ceramic slurry.

4.3 Alumina slurry preparation

Alumina aqueous slurry containing 20 and 25 vol% alumina has been prepared with the addition of deflocculent and binder. The particle size distribution of the alumina powder has been carried out using Melvern Particle size analyser. Darvan C 1 wt% is used as dispersant. 5 wt% PVA solutions have been used as binder. Requisite amount of alumina powder has been weighted using an electronic balance. The requisite amount of Darvan C and Binder solution has been taken in Teflon pot. Solvent water has of measured amount has been added to the pot. The binder, solvent and the dispersant was ball billed for 2 hrs before addition of the powder. After this initial homogenization alumina powder has been added to pot and milled for overnight. Thus aqueous slurry of alumina powder was prepared.

4.4 Characterization of slurry

4.4.1 Zeta Potential

Zeta potential is the potential difference between stern layer and dispersed layer. The value of zeta potential can be related to the stability of colloidal dispersions. The zeta potential shows the degree of repulsion between adjacent and similar charge particle in a suspension. A high zeta potential refers to stability or the suspension has resist to aggregation by sedimentation. When the potential is low then attraction exceeds repulsion and suspension becomes flocculated. So suspension with high zeta potential (negative or positive) is electrically stabilized while suspension with low zeta potential tends to coagulate. Zeta potential is measured by Zetasizer. The suspension comprises of different Darvan C content and zeta potential at different pH is measured.

4.4.2 Viscosity

Slurry with different Darvan C content has been prepared and the viscosity is measured at different shear rate. The measurement of viscosity is done by rotational viscometer instrument.

4.4.3 Sedimentation height

For measurement of sedimentation height the prepared slurry with different Darvan C content is poured it into a measuring cylinder and the height is measured as a function of time.

4.5 Green body fabrication

For fabrication of green body 65ml of the stable slurry was taken and poured it into pre-coated mould, rotated at 4900 rpm for different time periods i.e. 10 minutes, 15 minutes and 20 minutes. The flow chart for the fabrication of alumina tube by the designed experimental set up is presented in Fig.4.5.

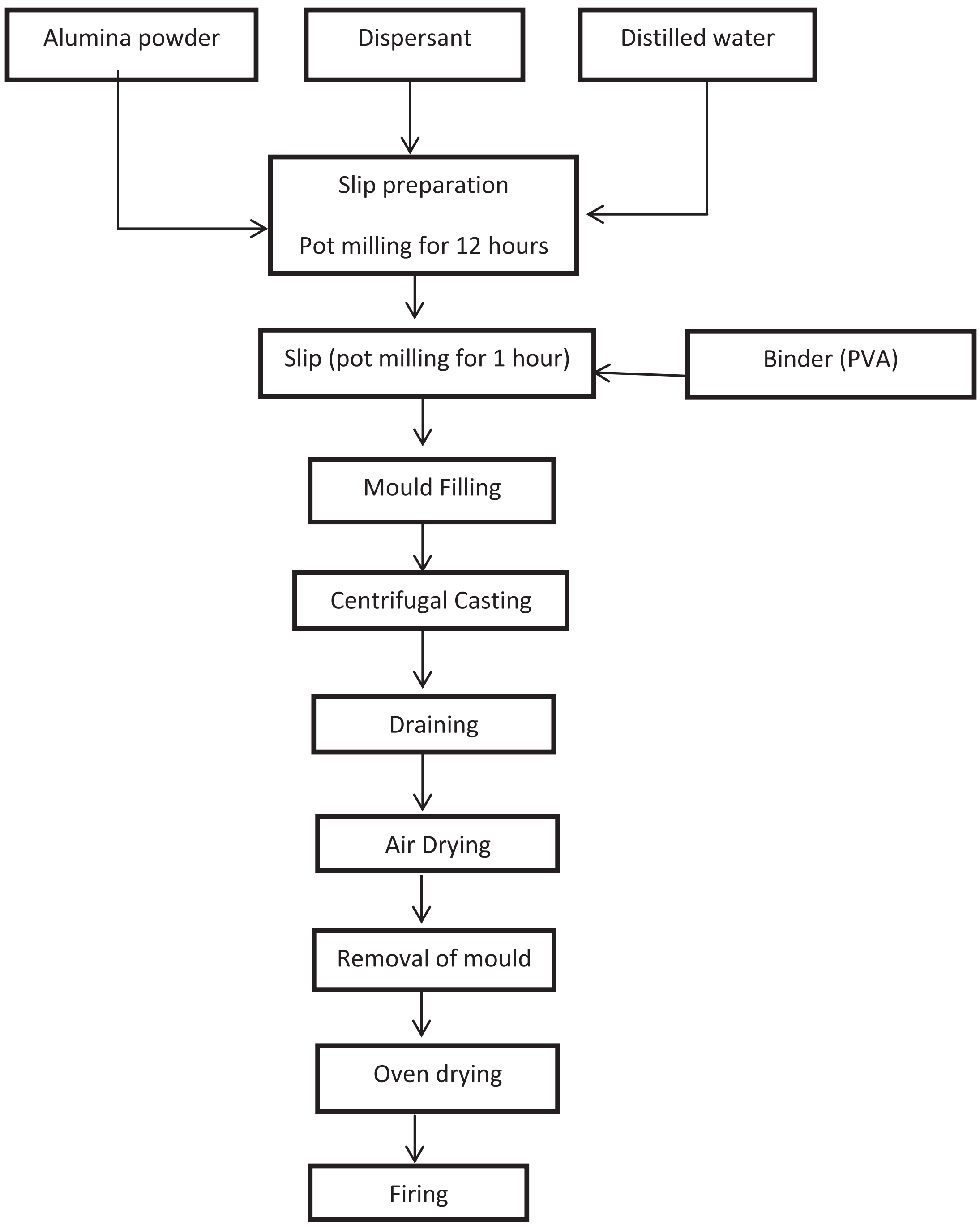


Fig. 4.5 Flow chart for the fabrication of alumina body using the deigned setup..

4.6 Sintering

Green body is fired at different temperature 1200^0C, 1350^0C, 1400^0C and 1650^0C with 2 hours of soaking time.

4.7 Microstructure

The surface characteristics of the sintered body were seen through a Scanning Electron Microscope (SEM) which was carried out in a secondary mode at 15kV.

CHAPTER-5

RESULT AND DISCUSSION

5.1 Zeta potential

Zeta potential measured as a function of pH and deflocculent concentration is presented in Fig. 5.1.

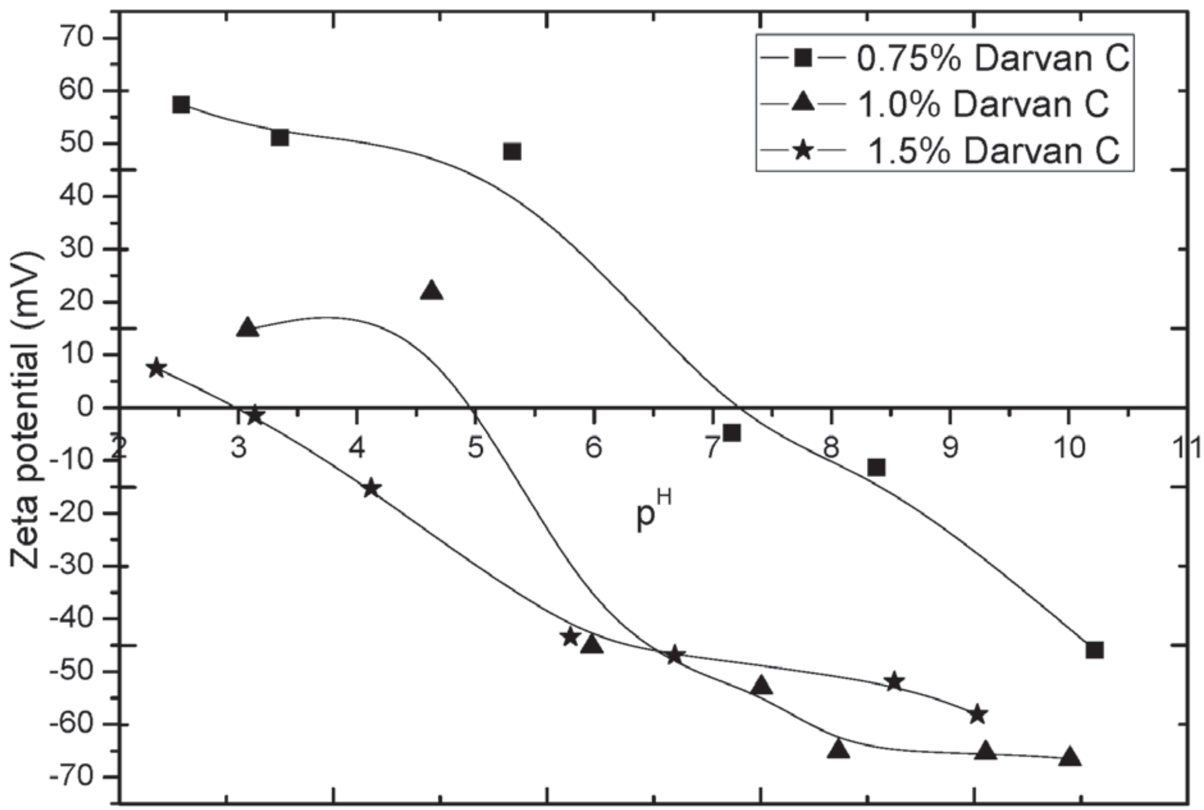


Fig. 5.1 Zeta potential of the alumina slurry as function of pH and Darvan C concentration.

The pH at which Zeta potential is found to be zero is called isoelectric point. It could be seen from the Fig.5.1 that the isoelectric point gradually decreases with increase in deflocculent content of the slurry. It could also be seen that at pH 7 or higher the zeta potential of the studied alumina powder increases with increase in Darvan C content in the slurry. Moreover the Zeta potential is negative indicates a negative surface charge on the alumina powder. In the pH range 8-9 it has been observed that zeta potential of the alumina suspension is higher, when the suspension is prepared with 1% Darvan C as compared to that prepared with 1.5%. This low value of the zeta potential in the ceramic slurry prepared with 1.5% Darvan C is attributed the over clouding of the electrolyte. Thus the present study suggest that alumina slurry prepared with 1% Darvan C is likely to provide more stability as the zeta potential is more than -30mV in the pH range 5-9.

5.2 Viscosity

Viscosity of the alumina slurry as a function of electrolyte concentration and shear rate is presented in Fig.5.2. It could be found that the viscosity of the slurry does not change appreciable with the shear rate in the studied shear rate. However, the viscosity of the slurry was found to decrease with incerase in electrolyte content of the slurry. The decrease in viscosity of the alumina slurry could be correlated the surface charge developed with the addition of the electrolyte into the slurry. The pH of the as prepared slurry was found to be in the range 5.6-6. It could be seen from the zeta potetial measurement curve that surface charge of the alumina increases with incerase in electrolyte concentration. The increase in zeta potention is attributed to the lowering of the viscosity with increase in electrolyte concentration.

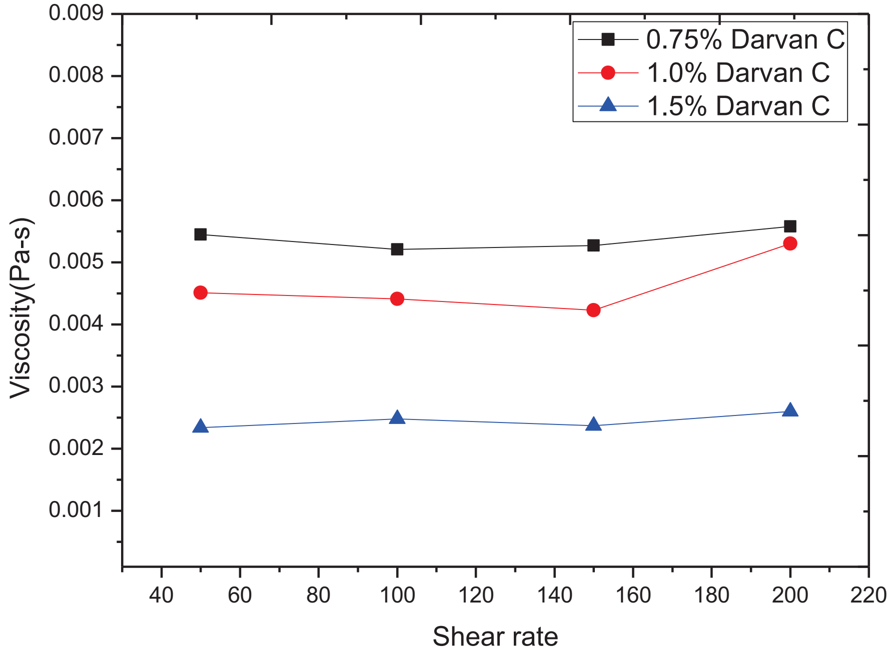


Fig. 5.2 Viscosity of the alumina slurry as function of Darvan C amount and shear rate.

5.3 Sedimentation height

The sedimentation height of the slurry as a function of time and electrolyte concentration has been shown in Fig.5.3. It could be seen form the figure that the sedimentation height of the slurry increases with increase in time, which is very obvious. The interesting feature is that the sedimentation height decreases with increase in electrolyte concentration in the slurry. This phenomenon could be correlated with the observed zeta potential and viscosity of the slurry discussed earlier. It is worthwhile to note that sedimentation does not change appreciable with the increase in Darvan C from 1.0% to 1.5%. Thus the study suggested that Darvan C 1% is the optimum amount to stabilize the studied alumina powder.

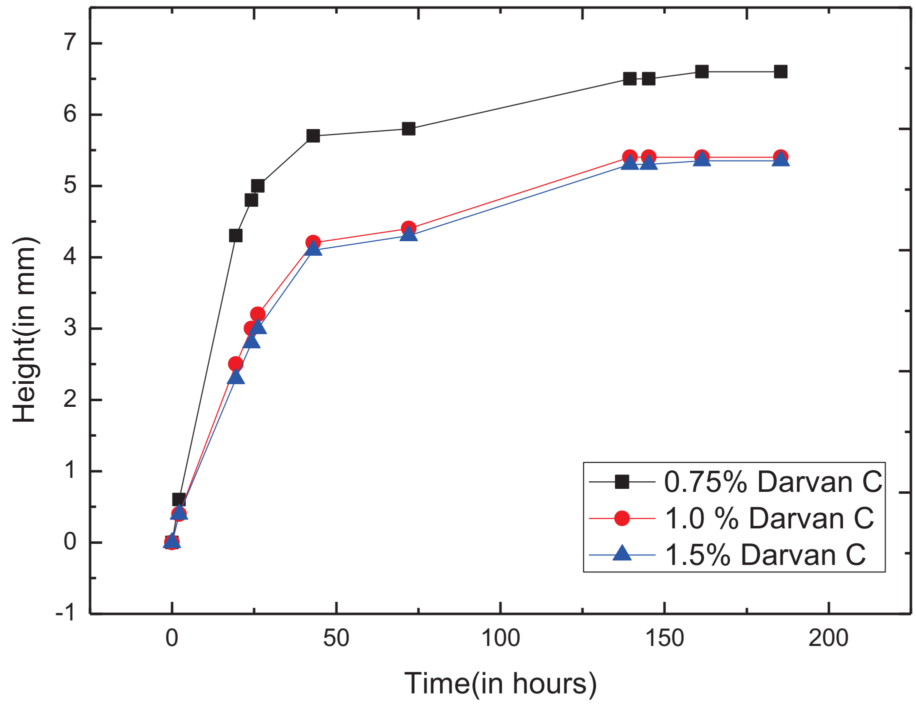


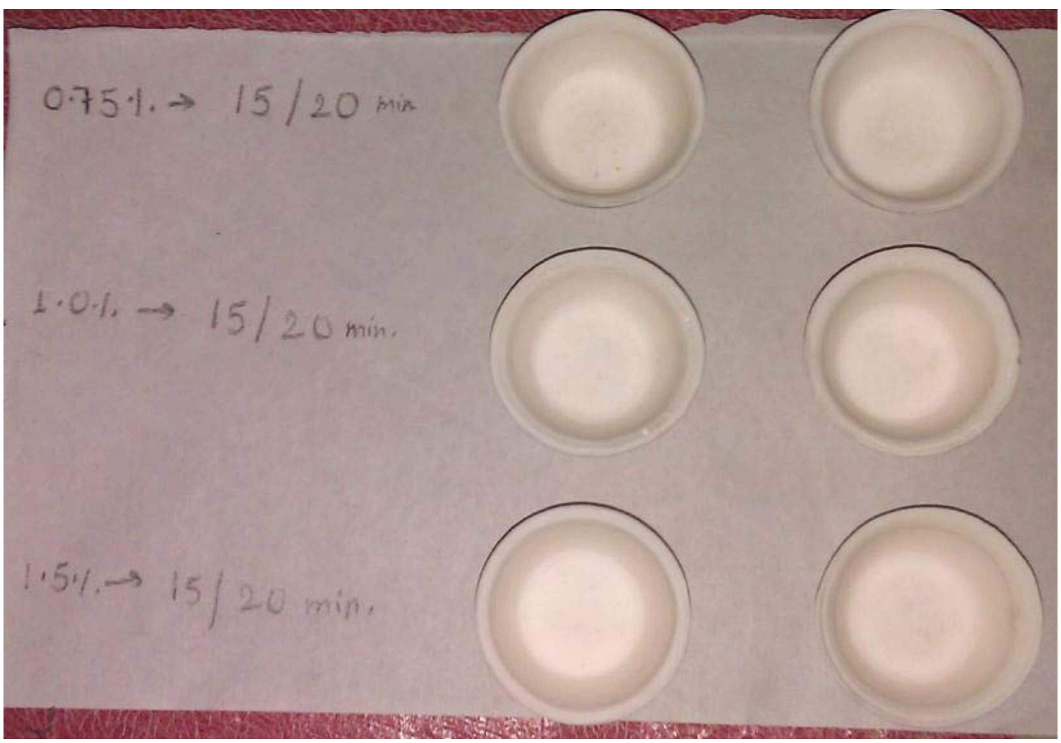
Fig.5.3 Sedimentation height as a function of time and Darvan C concentration of the slurry.

5.4 Green Cast body

Alumina tube fabricated with the designed experimental set up has been shown in Fig.5.4(a). It is interesting to note that hollow ceramic tube with smooth surface could be fabricated by this setup. However, it has also been noticed that the wall thickness of the cast body is very small. In order to increase the wall thickness an attempt has been made to study the effect of particle loading on the thickness of cast ware.

Alumina crucible fabricated by drain casting technique is shown in Fig.5.4(b). It has been observed that centrifugal cast body surface is smoother than drain cast body. It has been also observed that inner surface is highly dense of centrifugal cast body compare to drain cast body.

(a)



(b)

Fig. 5.4 Sintered alumina ceramics prepared by (a) centrifugal casting technique and (b) Drain casting technique

5.5 Effect of casting time

The weight of alumina retained in the supernatant waste of the centrifugal casting process has been studied as a function of particle loading in the slurry has been shown in Fig.5.5. It could be seen that the amount of alumina retained in the supernatant liquid decreases with the increase in casting time, which is obvious. It has also been seen that the alumina retention in the supernatant liquid decreases with increase in alumina loading in the slurry. As the concentration of alumina in the slurry increases the viscosity of the slurry is likely to increase which makes the deposition more effective as a result the retention of alumina in the supernatant decreases. Thus the study suggests that in order to achieve a thick cast layer by this technique high particle loading slurry will be preferred.

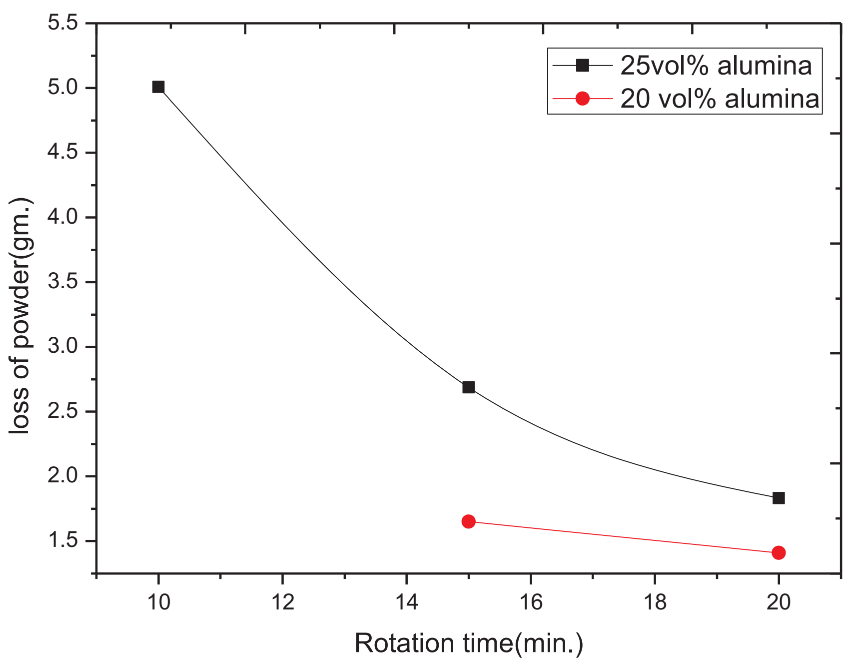
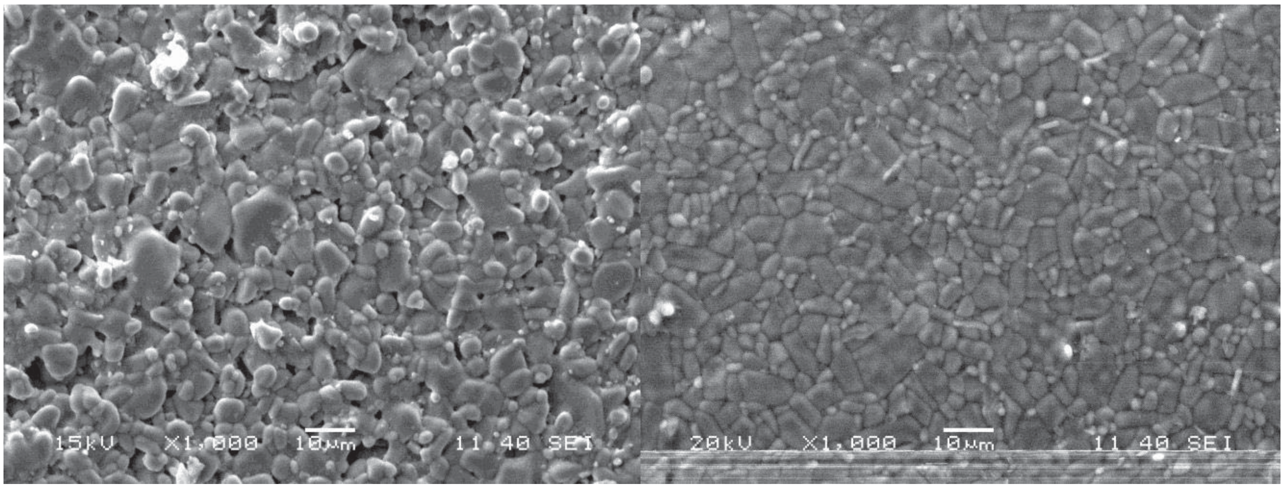


Fig. 5.5 Yield of alumina slurry during centrifugal casting as a function of casting time and solid loading

5.6 Microstructure

Fig.5.6 (a) and (b) shows the microstructure of inner surface of drain cast and centrifugal cast body respectively.



(a) **(b)**

Fig. 5.6 SEM micrographs of (a) drain cast and (b) centrifugal cast alumina body sintered at 1650^0C

Both are fired at same temperature 1650^0C with 2 hours soaking time. It has been observed that centrifugal cast body has highly dense compare with drain cast body. Moreover, the microstructure of the centrifugal cast body is more homogeneous as compared to that of drain cast body. There exists a differential centrifugal force among the particles present in slurry. The centrifugal forces on a large particle will be more as compared to the small particles. As the result the large particles are likely to consolidate quickly as compared to the small particle. Thus the microstructure of the cast body will have distrusted particles along the thickness of the cast layer. The outer layer will have large particles as compared to the inner surface which will has small particles only. The small particles due to their surface area will densify more leads to a more dense microstructure.

CHAPTER-6

CONCLUSION

The following conclusion could be achieved from the present study:

1. Experimental setup for centrifugal casting is successfully fabricated using laboratory centrifuge and design and fabrication of a suitable rotor head.
2. Alumina tube has been successfully fabricated using the fabricated setup from aqueous alumina slurry.
3. Centrifugal cast ceramic body likely to have graded microstructure. The inner surface will have high density as compared to the outer surface.

CHAPTER-7

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